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IS 1448-91 (1979): Methods of test for petroleum and its products, Part 91: Emulsion characteristics of petroleum oils and synthetic fluids [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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(Reaffirmed 2002)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 91]

EMULSION CHARACTERISTICS OF PETROLEUM OILS AND SYNTHETIC FLUIDS

UDC 665·6/·7 : 541·182·051·3

Adapted from ASTM Designation D 1401-67 (*Reapproved 1977*)

1. SCOPE

1.1 This method of test describes a procedure for the measurement of the ability of petroleum oils or synthetic fluids to separate from water.

NOTE 1 — Although developed specifically for steam-turbine oils having viscosities of 32 to 97 cSt at 38°C, this method may be used to test oils of other types having various viscosities and synthetic fluids. It is recommended, however, that the test temperature be raised to 82 ± 1°C when testing products more viscous than 97 cSt.

NOTE 2 — Other test temperatures such as 25°C may also be used.

NOTE 3 — When testing synthetic fluids whose relative densities are greater than that of water, the procedure is unchanged, but it should be noted that the water will probably float on the emulsion or liquid.

2. OUTLINE OF THE METHOD

2.1 A 40-ml sample and 40 ml of distilled water (*see* Note 1 under **6.1**) are stirred for 5 minutes at 54°C (*see* Notes under **1.1**) in a graduated cylinder. The time required for the separation of the emulsion thus formed is recorded. If complete separation does not occur after standing for 1 hour the volumes of oil (or fluid), water, and emulsion remaining at the time are reported.

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3. APPARATUS

3.1 Cylinder — 100-ml graduated from 5 to 100 ml in 1·0 ml divisions, made of heat resistant glass, or equivalent. The inside diameter shall be not less than 27 mm and not more than 30 mm throughout its length, measured from the top to a point 6 mm from the bottom of the cylinder. The overall height of the cylinder shall be 225 to 260 mm. The graduation shall not be in error by more than 1 ml at any point on the scale.

3.2 Heating Bath — Sufficiently large and deep to permit the immersion of at least two test cylinders in the bath liquid up to their 85 ml graduations. The bath shall be capable of being maintained at a temperature of 54 ± 1°C, and shall be fitted with clamps which hold the cylinder in a position so that the longitudinal axis of the paddle corresponds to the vertical centre line of the cylinder during the stirring operation. The clamps shall hold the cylinder securely while its contents are being stirred.

3.3 Stirring Paddle — Made of chromium plated or stainless steel and conforming to the following dimensions:

Length	120 ± 1·5 mm
Width	19 ± 0·5 mm
Thickness	1·5 mm

It is mounted on a vertical shaft of similar metal, approximately 6 mm in diameter, connected to a drive mechanism which rotates the paddle on its longitudinal axis at 1 500 ± 15 rpm. The apparatus is of such design that, when the cylinder is clamped in position and the paddle assembly is lowered into the cylinder, a positive stop engages and holds the assembly when the lower edge of the paddle is 6 mm from the bottom of the cylinder. During the operation of the stirrer, the centre of the bottom edge of the paddle shall not deviate more than 1 mm from the axis of rotation. When not in operation, the paddle assembly can be lifted vertically to clear the top of the graduated cylinder.

4. REAGENT

4.1 Cleaning Solvent — Light petroleum hydrocarbon solvent (*see* IS : 1745-1978*) for petroleum oils. Use other appropriate solvents for dissolving synthetic fluids.

5. PREPARATION OF APPARATUS

5.1 Clean the graduated cylinder by removing any film of oil (or fluid) with cleaning solvent followed by a wash first with acetone and then with

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*Specification for petroleum hydrocarbon solvents (*second revision*).

tap water. Immerse the cylinder in a cleaning solution of hot chromic acid. Rinse thoroughly with tap water and then with distilled water.

5.2 Clean the stirring paddle and shaft with absorbent cotton or tissue wet with cleaning solvent and air dry. Care shall be taken not to bend or misalign the paddle assembly during the cleaning operation.

6. PROCEDURE

6.1 Heat the bath liquid to 54 ± 1°C and maintain it at that temperature throughout the test. Add distilled water (*see* Notes 1 and 2) to the graduated cylinder up to the 40-ml mark and then add to the same cylinder a representative sample of the oil (or fluid) under test until the top level of the oil reaches the 80-ml mark on the cylinder. Place the cylinder in the bath and allow the contents to reach bath temperature. Normally this will require about 10 minutes.

NOTE 1 — If initial volumetric measurements are made at room temperature, expansion occurring at the elevated test temperature will have to be considered. For example, there will be a total volumetric expansion of about 2 to 3 ml at 82°C. Corrections to each volume reading at 82°C, therefore, should be made so that the total of the volume readings made for oils (or fluid), water, and emulsion does not exceed 80 ml. An alternative procedure which would avoid the corrections is to make the initial volumetric measurements at the test temperature.

NOTE 2 — A one-percent sodium chloride (NaCl) solution or artificial sea water shall be used in place of distilled water when testing certain oils or fuels used in marine applications. The artificial sea water shall have the following composition:

Salt	*g/litre*
$NaCl$	24·54
$MgCl_2.6H_2O$	11·10
Na_2SO_4	4·09
$CaCl_2$	1·16
KCl	0·69
$NaHCO_3$	0·20
KBr	0·10
H_3BO_3	0·03
$SrCl_2.6H_2O$	0·04
NaF	0·003

The solution can be conveniently prepared as follows:

The method avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using pure chemicals and distilled water, prepare the following stock solutions:

Stock Solution No. 1

$MgCl_2.6H_2O$	3 885 g
$CaCl_2$ (anhydrous)	406 g
$SrCl_2.6H_2O$	14 g

Dissolve and dilute to 7 litres.

Stock Solution No. 2

KCl	483 g
$NaHCO_3$	140 g
KBr	70 g
H_3BO_3	21 g
NaF	2·1 g

Dissolve and dilute to 7 litres.

To prepare the artificial sea water, dissolve 245·4 g of NaCl and 40·94 g of Na_2SO_4 in a few litres of distilled water, add 200 ml of Stock Solution No. 1 and 100 ml of Stock Solution No. 2 and dilute to 10 litres. Stir the 10 litres batch and add 0·1 N Na_2CO_3 until the *p*H is between 7·8 and 8·2. One or two millilitres of the carbonate solution will be required.

6.2 Clamp the cylinder in place directly under the stirring paddle. Lower the paddle into the cylinder until the stop engages at the required depth. Start the stirrer and a stop watch simultaneously and adjust the stirrer, as required, to a speed of 1 500 ± 15 rpm. At the end of 5 minutes, stop the stirrer and raise the stirring assembly until it is just clear of the graduate. Wipe the paddle with a policeman (*see* Note), allowing the liquid thus removed to drop back into the cylinder. Remove the cylinder from the retaining clamps and transfer it carefully to another section of the bath. At 5 minutes intervals lift the cylinder out of the bath, inspect, and record the volumes of the oil (or fluid), water, and emulsion layers.

NOTE — The policeman should be made of material resistant to the oil or fluid.

7. REPORT

7.1 Record the time (at 5 minutes intervals) required for the emulsion to be reduced to 3 ml or less. If the emulsion is more than 3 ml one hour after the end of the stirring period, discontinue the test and record the amounts, in millilitres, of oil, water, and emulsion remaining (*see* Note under **7.2**). For uniformity, report the test results in the manner shown in the following examples:

40-40-0 (20)	Complete separation occurred in 20 minutes. More than 3 ml of emulsion had remained at 15 minutes.

40-37-3 (20)	Complete separation had not occurred, but the emulsion reduced to 3 ml so the test was ended.
39-35-6 (60)	More than 3 ml of emulsion remained after 60 minutes—39 ml of oil, 35 ml of water, and 6 ml of emulsion.

7.2 The appearance of each layer may be described in the following terms.

7.2.1 *Oil (or Fluid) Layer*

7.2.1.1 *Clear*

7.2.1.2 *Hazy* (*see* Note 2)

7.2.1.3 *Cloudy* (*or milky*) (*see* Note 2)

7.2.2 *Water Layer*

7.2.2.1 *Clear*

7.2.2.2 *Lacy or bubbles present or both*

7.2.2.3 *Hazy* (*see* Note 2)

7.2.2.4 *Cloudy* (*or milky*) (*see* Note 2)

7.2.3 *Emulsion*

7.2.3.1 *Loose and lacy*

7.2.3.2 *Cloudy* (*or milky*) (*see* Note 3)

7.2.3.3 *Creamy* (*like mayonnaise*) (*see* Note 3)

NOTE 1 — Certain oils may produce a hazy oil layer. In situations where the measurement of the oil and water layer indicates essentially complete separation, the upper layer shall be reported as oil.

NOTE 2 — A hazy layer is defined as being translucent and a cloudy layer opaque.

NOTE 3 — The principal difference between cloudy and creamy emulsions is that the former is quite fluid and probably unstable while the latter has a thick consistency and is probably stable. A cloudy emulsion will readily flow from an inclined graduate while a creamy emulsion will not.

7.3 The appearance of the oil/emulsion and water/emulsion interfaces may be described in the following terms.

7.3.1 *Well-Defined, Sharp*

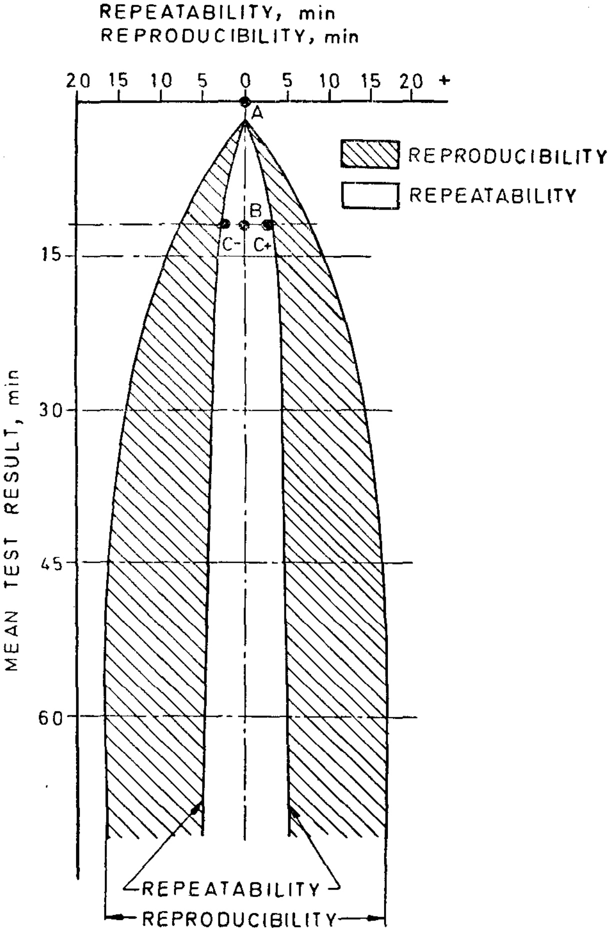
7.3.2 *Ill-Defined, Bubbles*

7.3.3 *Ill-Defined, Lace*

7.4 Report the test temperature if other than 54°C and the aqueous medium if other than distilled water.

8. PRECISION

8.1 The precision of this test was obtained on steam-turbine oils having viscosities of 32 to 97 cSt at 38°C and is expressed graphically in Fig. 1. The graph shows the maximum allowable deviation in minutes for repeatability and reproducibility (95 percent confidence) from mean emulsion test results for these oils. It may not be applicable to other oils or fluids.



Use of Chart — Calculate the mean test result in minutes. Enter chart at the zero point, *A* on the ordinate and move to the right on the abscissa to point *B*. Compute and locate the deviation points *C*+ and *C*−, from the mean test result. If the deviation points fall within the repeatability area, then the results are within the precision of the test.

Example — An oil has emulsion values of 40-40-0 (10 minutes) and 40-40-0 (15 minutes). The mean test result is 12·5 min (*B*) and the deviation from the mean is +2·5(*C*+) and −2·5(*C*−).

These points fall within the repeatability area.

Use this graph similarly for the reproducibility of means of different laboratories.

FIG. 1 CHART FOR DETERMINING TEST PRECISION

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